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Key indicators

Single-crystal X-ray study
 $T = 173\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.029
 wR factor = 0.074
 Data-to-parameter ratio = 16.7

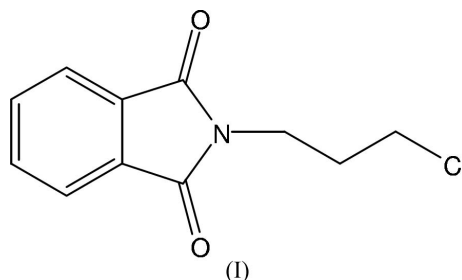
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

2-(3-Chloropropyl)-2,3-dihydro-1*H*-isoindole-1,3-dione

The geometric parameters of the title compound, $\text{C}_{11}\text{H}_{10}\text{ClNO}_2$, are in the normal ranges. The phthalimide moiety is planar and the chloropropyl chain adopts a synclinal conformation. The crystal packing is stabilized by two intermolecular $\text{C}-\text{H}\cdots\text{O}$ contacts.

Comment

The title compound, (I), also named *N*-(3-chloropropyl)-phthalimide, is used as an intermediate for the synthesis of biologically active heterocycles (Kerrigan *et al.*, 2000; Salvati *et al.*, 2005). In view of its importance and in order to determine the conformation of this molecule, a crystal structure determination has been carried out.



A perspective view of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal [Cambridge Structural Database (CSD), Version 1.7; *MOGUL*, Version 1.0.1; Allen, 2002]. The isoindole-1,3-dione system is planar (r.m.s. deviation = 0.011 Å). Methylene atom C9 attached to the N atom deviates from this plane by only 0.031 (1) Å. The chloropropyl moiety adopts a synclinal conformation [$\text{Cl1}-\text{C11}-\text{C10}-\text{C9} = -67.71(12)^\circ$]. This conformation is also found for seven out of eight structures containing the $\text{Cl}-\text{CH}_2\text{CH}_2\text{CH}_2-\text{AA}$ fragment (AA = any atom) retrieved from the CSD. The only

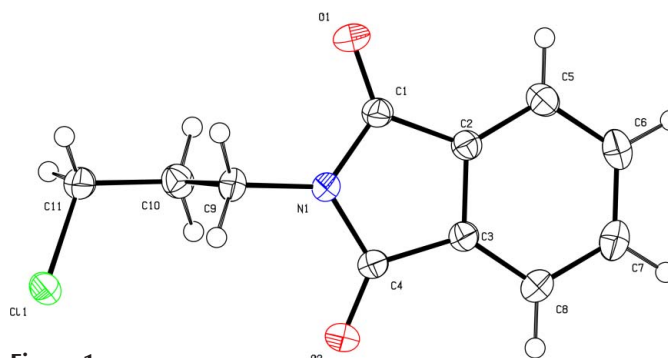


Figure 1
 Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level.

structure not showing this conformation is 8-amino-7-(3-chloropropyl)theophylline benzene solvate, with a torsion angle of 175.8° (Karczmarzyk & Pawlowski, 1998). The $-\text{CH}_2\text{CH}_2\text{CH}_2-\text{N}$ chain, on the other hand, adopts an anti-periplanar conformation [$\text{C11}-\text{C10}-\text{C9}-\text{N1} = 177.15(10)^\circ$]. The crystal packing is stabilized by two intermolecular $\text{C}-\text{H}\cdots\text{O}$ contacts (Table 2).

Experimental

A mixture of isoindole-1,3-dione (1.47 g, 10 mmol), anhydrous potassium carbonate (1.38 g, 10 mmol) and 1-bromo-3-chloropropane (1.57 g, 10 mmol) was stirred at room temperature in dimethylformamide (10 ml) for 6 h to give the title compound, which was recrystallized from methanol (m.p. 340 K).

Crystal data

$\text{C}_{11}\text{H}_{10}\text{ClNO}_2$	$D_x = 1.393 \text{ Mg m}^{-3}$
$M_r = 223.65$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 29 481 reflections
$a = 4.5421(4) \text{ \AA}$	$\theta = 3.7\text{--}27.1^\circ$
$b = 15.3996(15) \text{ \AA}$	$\mu = 0.34 \text{ mm}^{-1}$
$c = 15.3471(13) \text{ \AA}$	$T = 173(2) \text{ K}$
$\beta = 96.605(7)^\circ$	Rod, colourless
$V = 1066.35(17) \text{ \AA}^3$	$0.38 \times 0.22 \times 0.22 \text{ mm}$
$Z = 4$	

Data collection

Stoe IPDS-II two-circle diffractometer	2290 independent reflections
ω scans	2103 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	$R_{\text{int}} = 0.033$
$T_{\text{min}} = 0.883$, $T_{\text{max}} = 0.930$	$\theta_{\text{max}} = 26.9^\circ$
11 334 measured reflections	$h = -5 \rightarrow 5$
	$k = -19 \rightarrow 19$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 0.3497P]$
$R[F^2 > 2\sigma(F^2)] = 0.029$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.074$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
2290 reflections	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$
137 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.064 (5)

Table 1

Selected bond lengths (\AA).

$\text{N1}-\text{C1}$	1.4001 (15)	$\text{C1}-\text{O1}$	1.2165 (14)
$\text{N1}-\text{C4}$	1.4015 (14)	$\text{C4}-\text{O2}$	1.2183 (15)
$\text{N1}-\text{C9}$	1.4660 (14)	$\text{C11}-\text{C11}$	1.8198 (13)

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C11}-\text{H11A}\cdots\text{O2}^i$	0.99	2.57	3.3251 (15)	133
$\text{C11}-\text{H11B}\cdots\text{O1}^{\text{ii}}$	0.99	2.43	3.2494 (15)	140

Symmetry codes: (i) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (ii) $2 - x, 1 - y, 1 - z$.

All H atoms were located in a difference map, but were then geometrically positioned and refined with fixed individual displacement parameters (set at 1.2 times U_{eq} of the parent atom) using a riding model, with $\text{C}-\text{H} = 0.95$ and 0.99 \AA for aromatic and methylene H atoms, respectively.

Data collection: *X-Area* (Stoe & Cie, 2001); cell refinement: *X-Area*; data reduction: *X-Area*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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